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WAR DEPARTMENT TECHNICAL BULLETIN

PROCEDURE FOR DETERMINING SERVICEABILITY OF CHLORINATED PARAFFIN STORED AT THEATER OF OPERATIONS DEPOTS

War Department, Washington 25, D. C. January 1945

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- 1. PURPOSE.** The purpose of this Technical Bulletin is to provide a method for determining serviceability of chlorinated paraffin (binder) stored in drums at theater of operations depots.
- 2. EQUIPMENT.** Necessary equipment for this procedure consists of the following:
 - a. Sampling thief (longer than a storage drum, with $\frac{1}{4}$ -inch inside diameter).
 - b. Two-liter, glass-stoppered bottles.
 - c. Laboratory equipment, reagents, and standard solutions for analytical method described in paragraph 5b.
- 3. FREQUENCY OF SURVEILLANCE INSPECTION.** Each lot of the material should be inspected at time intervals as follows, depending on the temperature zone in which it is stored:

Mean shade temperature in hottest month	Frequency of inspection in months
Over 90° F.....	6
70° F. to 90° F.....	9
Under 70° F.....	12

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4. **SAMPLES.** *a. Sample drums.* A number of representative drums should be selected at random from a lot as indicated in the following table:

Number of drums in lot	Number of sample drums required
Up to 8	2.
9 to 27	3.
28 to 64	4.
65 to 125	5.
126 to 215	6.
216 to 499	8.
500 or more	9 plus 1 for each 500 or portion thereof in excess of 1,000.

b. Composite sample. Each sample drum should be rolled, prior to withdrawing a representative liquid sample, for a time interval as follows:

Average temperature of drum contents	Rolling time (minutes)
70° F. or over	5
60° F. to 69° F. (incl.)	10
50° F. to 59° F. (incl.)	15

If contents of the drum are under 50° F., it should be kept in a warm room for at least 24 hours prior to sampling. Sample is taken, using a glass or metal sampling thief of suitable diameter (par. 2a), to provide a sample volume of approximately 100 cubic centimeters, tube being lowered slowly to reach bottom of drum. These liquid samples from a given lot are combined as a composite sample which is stored in a properly labeled, glass-stoppered bottle.

5. **SURVEILLANCE INSPECTION.** *a. Visual examination of sample drums.* Each sample drum should be inspected visually, at the time contents are sampled, for faults noted in accompanying suggested report form, items 15 and 17. These observations are intended to disclose minor defects, none of which would render the chlorinated paraffin unserviceable.

b. Test for hydrochloric acid. Each composite sample should be tested for acidity, calculated as HCl, by the following method:

(1) Transfer 20 milliliters (ml) of sample into a 250-ml separatory funnel, add 50 ml of benzene, and mix thoroughly by shaking.

(2) Add 30 ml of 95-percent ethyl alcohol to the mixture, shake, add 100 ml distilled water, and mix thoroughly by shaking.

(3) Allow mixture to stand until layers separate.

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(4) Separate alcohol-water solution and add a few drops of mixed indicator solution, consisting of 0.050 gram of methyl red and 0.075 gram of bromocresol green (tetrabromo-m-cresol sulfonphthalein) dissolved in 100 ml of 95-percent ethyl alcohol.

(5) Titrate with 0.020 N (Normal) NaOH (sodium hydroxide) to the intermediate purple end-point.

(6) Run a blank, using the same volumes of benzene, alcohol, and water, but no sample, and titrate in the same manner.

(7) Formula for above procedure is:

(ml NaOH ml blank) N.F. 36.5 mg HCl per 20 ml of sample. (N.F.—normality factor of NaOH solution.)

c. *Recording of data.* A suggested form for the accumulation of surveillance data is included in this Technical Bulletin.

6. SERVICEABILITY QUALIFICATIONS. a. A lot of chlorinated paraffin in drums should be considered provisionally unserviceable for use provided item number 21 of suggested report form is answered in the negative. Any drum which is obviously in a bad exterior condition which might soon lead to drum failure should be removed at once.

b. It is suggested that a 1-quart sample of chlorinated paraffin in a sealed and labeled glass-stoppered bottle, corresponding to each composite sample found provisionally unserviceable, be submitted to Chief, Technical Division, Office of the Chief, Chemical Warfare Service, Edgewood Arsenal. Each sample should be accompanied by pertinent surveillance data.

SUGGESTED REPORT FORM SURVEILLANCE DATA

A. Identification characteristics

1. Date inspected					
2. Lot number					
3. Batch number					
4. Contract number					
5. Manufacturer's name or initials					
6. Month and year filed					

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B. Pertinent depot information

7. Designation of depot					
8. Number containers in lot					
9. Date received at depot					
10. Date last surveillance inspection					
11. Indicate by check which of following storage conditions apply:					
a. In covered trenches or better					
b. Under tarpaulins					
c. Exposed to sunshine					
12. Mean shade temperature at depot during hottest month					
13. Mean annual rainfall at depot					

C. Container characteristics

14. Size of drums, gallons					
15. Number drums in lot without white heads					
16. Number drums structurally satisfactory					
17. Indicate by check marks which faults are present and number drums with each, in parentheses					
a. Leaks					
b. External corrosion					
(1) Excessive (over 25 percent area)					
(2) Moderate (5-25 percent area)					
(3) Some (under 5 percent area)					
c. Name other structural defects, if any					

D. Physical and chemical characteristics

18. Material contains solid matter (yes or no)					
19. Solid matter (if any) dissolves entirely on heating sample to 60 C. (yes or no) ¹					
20. HCl contents, percent (provisional limit=0.02 percent)					

¹ Acceptable only if answer is yes.

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E. Miscellaneous information

21. Material considered serviceable (yes or no) ²					
22. Officer supervising inspection					
Name					
Rank					
23. Responsible depot chemical officer					
Name					
Rank					
24. Remarks					

² Note—Yes, if items 19 and 20 are within limits.

[AG 300.5 (28 Dec 44)]

BY ORDER OF THE SECRETARY OF WAR:

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For explanation of symbols, see FM 21-6.